

Data Limitations and Validation Report

for Lockheed Idaho Technologies

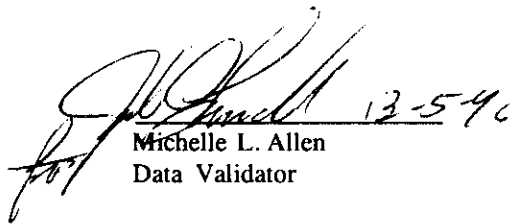
Case No. 93030806, SDG 93030806

Argonne National Laboratory - West

TCL Appendix IX Volatile Organics

Four Aqueous Samples

Validated by:

 13-5-96
Michelle L. Allen
Data Validator

Approved by:

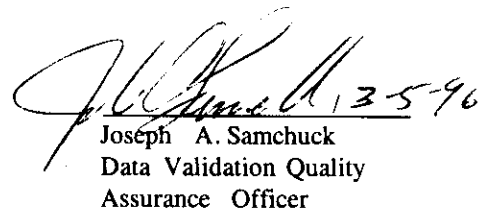
 13-5-96
Joseph A. Samchuck
Data Validation Quality
Assurance Officer

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1.0 INTRODUCTION

The Argonne National Laboratory - West sample set for Case No. 93030806, SDG 93030806 consists of four aqueous samples analyzed for Target Compound List (TCL) Appendix IX volatile organic compounds. The analyses were performed by Biospherics, Inc. using the protocols outlined in the "Analytical Laboratory Services for Environmental Groundwater Samples for the Argonne National Laboratory - West" Statement of Work (SOW). A total of 184 TCL sample data points were reported in this analytical data set.

The analytical data from these analyses were reviewed by HALLIBURTONNUS Corporation personnel as Level A in accordance with ERP Standard Operating Procedure SMO-SOP-12.1.3.

2.0 QUALITY CONTROL SUMMARY

The data were evaluated based on the following parameters:

- Data Completeness
- Holding Times
- GC/MS Tuning and Mass Calibration
- Initial and Continuing Calibrations
- Blank Analyses
- * Surrogate Spike Recoveries
- * Matrix Spike/Matrix Spike Duplicate Results
- Internal Standards Performance
- * System Performance and Detection Limits
- Compound Quantitation/Identification
- Laboratory Performance

The asterisk indicates that all quality control criteria were met for this parameter. Problem areas affecting data usability are discussed in Section 4.0 of this report.

3.0 DATA COMPLETENESS

The data presented in Case No. 93030806, SDG 93030806 consists of TCL Appendix IX volatile organic results for four (4) aqueous sample as follows:

93030806-1 (MW-11)	93030806-2 (EBR II NO1)
93030806-3 (EBR II NO2)	93030806-4 (TRIP BLANK)

The Chain-of-Custody (C-O-C) form for the above samples was not present in the data package. In addition, the data package was missing a laboratory method blank and the raw data (quantitation report, chromatogram, etc.) for sample 93030806-3 (EBR II NO2). Hence, the results reported for this sample could not be confirmed by the data validator. It should be noted that the presentation and documentation of the data package deliverables were extremely poor. The data package does not conform to a Level A deliverable.

4.0 SUMMARY OF DATA USABILITY

The Chain-of-Custody (C-O-C) form was not included in the data package or with the C-O-Cs received with this project. Hence, the holding times from sampling to analysis could not be evaluated for the samples contained in this SDG.

The bromofluorobenzene (BFB) tune (Form V) contained erroneous % relative abundances. The data reviewer amended this form.

The Percent Relative Standard Deviations (%RSDs) and Relative Response Factors (RRFs) reported for the target compounds on the initial calibration Form VI were not correct. For example, the laboratory reported nominal concentrations on the Form VI instead of the RRFs. Hence, the samples could not be evaluated for these parameters.

Incorrect internal standard areas/retention times were reported for the 12 hour standard on the laboratory Form VIII. In addition, the sample internal standard areas and retention times were not present on this form. Therefore, the samples were not evaluated for internal standard area and corresponding retention time criteria.

The Appendix IX volatile compounds acrylonitrile, 2-chloroethylvinyl ether, methyl-tert-butyl ether, allyl chloride, methacrylonitrile, propanenitrile, and methyl methacrylate were not included in the initial calibration standards. Hence, these compounds were rejected on the environmental sample Form Is.

Acetone and m/p-xylenes were detected in sample 93030806-4 (TRIP BLANK), however, the laboratory did not report these compounds on the sample Form I. No action was taken since these compounds were detected below the detection limit.

Annotated laboratory Form I data summary reports showing the data and relevant qualifier flags applied are presented in Appendix A of this report. Copies of the unqualified data summary reports as reported by the laboratory are provided in the attached Appendix B. The attached Appendix C includes documentation to support the findings discussed in this report.

A sample-specific summary of the data validation flags applied is depicted in Table 1, appearing on the following page.

4.1 Holding Times

The C-O-C form associated with the samples in this SDG was not present in the data package. Therefore, the holding time from sampling until analysis could not be evaluated by the data validator for the samples in this SDG.

4.2 Calibrations

The initial calibration Form VI contained incorrect %RSDs and RRFs for the target compounds. The samples were not evaluated for these parameters.

4.3 Internal Standards

The laboratory Form VIII had incorrect internal standard areas/retention times for the 12 hour standard. In addition, the environmental sample internal standard areas/retention times were not present on this form. Hence, the samples were not evaluated for these quality control parameters.

4.4 Compound Quantitation/Identification

Upon review of the sample raw data, acetone and m/p-xylenes were detected in sample 93030806-4 (TRIP BLANK). However, the laboratory did not report these results on the sample Form I. No action was taken by the data validator since these results were reported below the detection limit.

TABLE 1

Lockheed Idaho Technologies

Argonne National Laboratory - West

Case No. 93030806, SDG 93030806

TCL Appendix IX Volatile Organics

Sample No.	Qualifier Flags
<hr/>	
93030806-1 (MW-11)	R ¹
93030806-2 (EBR II NO1)	R ¹
93030806-3 (EBR II NO2)	R ¹
93030806-4 (TRIP BLANK)	R ¹

5.0 SUMMARY OF LABORATORY PERFORMANCE

The raw data associated with sample 93030806-3 (EBR II NO2), the C-O-C form, and a laboratory method blank were missing from this SDG. The sample numbers (as indicated on the sample Form Is) were not used to name the environmental samples referenced in this data package due to the repetition of these sample IDs used in additional SDGs associated with this project (i.e., "EBR II NO1, EBR II NO2, M-11, and TRIP BLANK"). Hence, the data validator used both Biospherics laboratory IDs, 93030806-1, -2, -3, and -4, and the field IDs. It should be noted that several forms had incomplete and/or incorrect times and dates, and the initial calibration Form VI and internal standard Form VIII were completely incorrect. The data validator attempted to manually correct some of these errors. The laboratory failed to standardize for several target compounds, hence, these were rejected. Some of the pages in the data package were not legible due to poor copy quality. The laboratory failed to report positive results for acetone and m/p-xylenes detected in one sample below the detection limit.

The overall documentation and completeness of the data package deliverable were extremely poor. The presentation of the information in this package compromises the validation review.

6.0 GLOSSARY OF DATA VALIDATION FLAGS

The following data validation flags were applied to the sample data for reasons detailed previously in this report:

R ¹	-	Reject, "R", nondetected results for acrylonitrile, 2-chloroethylvinyl ether, methyl-tert-butyl ether, allyl chloride, methacrylonitrile, propanenitrile, and methyl methacrylate as a result of the laboratory's failure to standardize for these compounds.
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7.0 REFERENCES

The data referenced in this report were validated in accordance with the protocols outlined in ERP Standard Operating Procedure SMO-SOP-12.1.3 as presented in ERP-SOW-37. In addition, details stipulating laboratory procedures as outlined in "Analytical Laboratory Services for Environmental Groundwater Samples for the Argonne National Laboratory - West" SOW were referenced.

APPENDIX A
QUALIFIED LABORATORY RESULTS

1A
VOLATILE ORGANIC ANALYSIS DATA SHEET

SAMPLE NO.

MW-11

Lab Name: BIOSPHERICS INCORPORATED Contract: ARGONNE

Lab Code: MW-11 Case No. 93030806 SAS No.: _____ SDG No.: _____

Matrix: (soil/water) WATER Lab Sample ID: 93030806-1

Sample wt/vol: _____ (g/mL) Lab File ID: 8240

Level: (low/med) LOW Date Received: 03/03/93

% Moisture: not dec. _____ Date Analyzed: 03/09/93 ¹⁰ _{3/13/96} m.d.

GC Column: _____ ID: _____ (mm) Dilution Factor: 1

Soil Extract Volume: _____ (uL) Soil Aliquot Volume: _____ (uL)

CAS NO. COMPOUND CONCENTRATION UNITS:
(ug/L or ug/Kg) UG/L Q

74-87-3-	Chloromethane	10 ^U
74-83-9-	Bromomethane	10 ^U
75-01-4-	Vinyl Chloride	10 ^U
75-00-3-	Chloroethane	10 ^U
75-09-2-	Methylene Chloride	5 ^U
67-64-1-	Acetone	10 ^U
75-15-0-	Carbon Disulfide	5 ^U
75-35-4-	1,1-Dichloroethene	5 ^U
75-34-3-	1,1-Dichloroethane	5 ^U
540-59-0-	1,2-Dichloroethene (total)	NR
67-66-3-	Chloroform	5 ^U
107-06-2-	1,2-Dichloroethane	5 ^U
78-93-3-	2-Butanone	100 ^U
71-55-6-	1,1,1-Trichloroethane	5 ^U
56-23-5-	Carbon Tetrachloride	5 ^U
75-27-4-	Bromodichloromethane	5 ^U
78-87-5-	1,2-Dichloropropane	5 ^U
10061-01-5-	cis-1,3-Dichloropropene	5 ^U
79-01-6-	Trichloroethene	5 ^U
124-48-1-	Dibromochloromethane	5 ^U
79-00-5-	1,1,2-Trichloroethane	5 ^U
71-43-2-	Benzene	5 ^U
10061-02-6-	trans-1,3-Dichloropropene	5 ^U
75-25-2-	Bromoform	5 ^U
108-10-1-	4-Methyl-2-Pentanone	10 ^U
591-78-6-	2-Hexanone	10 ^U
127-18-4-	Tetrachloroethene	5 ^U
79-34-5-	1,1,2,2-Tetrachloroethane	5 ^U
108-88-3-	Toluene	5 ^U
108-90-7-	Chlorobenzene	5 ^U
100-41-4-	Ethylbenzene	5 ^U
100-42-5-	Styrene	5 ^U
1330-20-7-	Xylene (total)	5 ^U

1A
VOLATILE ORGANIC ANALYSIS DATA SHEET

SAMPLE NO.

Lab Name: BIOSPHERICS INCORPORATED Contract: ARGONNE

MW-11

Lab Code: MW-11 Case No. 93030806 SAS No.: _____ SDG No.: _____

Matrix: (soil/water) WATER Lab Sample ID: 93030806-1

Sample wt/vol: _____ (g/mL) _____ Lab File ID: 8240

Level: (low/med) LOW Date Received: 03/03/93

% Moisture: not dec. _____ Date Analyzed: 03/09/93 ¹⁰ *MLT*

GC Column: _____ ID: _____ (mm) Dilution Factor: 1 *3/18/96*

Soil Extract Volume: _____ (uL) Soil Aliquot Volume: _____ (uL)

CAS NO. COMPOUND CONCENTRATION UNITS:
(ug/L or ug/Kg) UG/L Q

97-63-2-	Ethyl Methacrylate	5 U	
107-02-8-	Acrolein	50 U	
108-05-4-	Vinyl Acetate	10 U	
107-13-1-	Acrylonitrile	50 U	<i>MLT</i>
110-75-8-	2-Chloroethyl Vinyl Ether	10 U	<i>3/3/96</i>
108-38-3-	M & P Xylene	10 U	
95-47-6-	O Xylene	5 U	
1634-04-4-	Methyl tert-Butyl Ether	5 U	
17-05-1-	Allyl Chloride	50 U	
126-98-7-	Methacrylonitrile	50 U	
107-12-0-	Propanenitrile	50 U	
74-88-4-	Iodomethane	50 U	
80-62-6-	Methyl Methacrylate	20 U	<i>V</i>

FORM I VOA

Form by WinLabChem (707) 864-0848/p/n11014/v3.0/1/1/94
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1A
VOLATILE ORGANIC ANALYSIS DATA SHEET

SAMPLE NO.

EBR II NO1

Lab Name: BIOSPHERICS INCORPORATED Contract: ARGONNE

Lab Code: EBRII NO1 Case No. 93030806 SAS No.: _____ SDG No.: _____

Matrix: (soil/water) WATER Lab Sample ID: 93030806-2

Sample wt/vol: _____ (g/mL) Lab File ID: 8240

Level: (low/med) LOW Date Received: 03/03/93

% Moisture: not dec. _____ Date Analyzed: 03/09/93

GC Column: _____ ID: _____ (mm) Dilution Factor: 1

Soil Extract Volume: _____ (uL) Soil Aliquot Volume: _____ (uL)

CAS NO. COMPOUND CONCENTRATION UNITS:
(ug/L or ug/Kg) UG/L Q

74-87-3-	Chloromethane	10U	
74-83-9-	Bromomethane	10U	
75-01-4-	Vinyl Chloride	10U	
75-00-3-	Chloroethane	10U	
75-09-2-	Methylene Chloride	5U	
67-64-1-	Acetone	10U	
75-15-0-	Carbon Disulfide	5U	
75-35-4-	1,1-Dichloroethene	5U	
75-34-3-	1,1-Dichloroethane	5U	
540-59-0-	1,2-Dichloroethene (total)	5U	
67-66-3-	Chloroform	NR	
107-06-2-	1,2-Dichloroethane	5U	
78-93-3-	2-Butanone	5U	
71-55-6-	1,1,1-Trichloroethane	100U	
56-23-5-	Carbon Tetrachloride	5U	
75-27-4-	Bromodichloromethane	5U	
78-87-5-	1,2-Dichloropropane	5U	
10061-01-5-	cis-1,3-Dichloropropene	5U	
79-01-6-	Trichloroethene	5U	
124-48-1-	Dibromochloromethane	5U	
79-00-5-	1,1,2-Trichloroethane	5U	
71-43-2-	Benzene	5U	
10061-02-6-	trans-1,3-Dichloropropene	5U	
75-25-2-	Bromoform	5U	
108-10-1-	4-Methyl-2-Pentanone	5U	
591-78-6-	2-Hexanone	10U	
127-18-4-	Tetrachloroethene	10U	
79-34-5-	1,1,2,2-Tetrachloroethane	5U	
108-88-3-	Toluene	5U	
108-90-7-	Chlorobenzene	5U	
100-41-4-	Ethylbenzene	5U	
100-42-5-	Styrene	5U	
1330-20-7-	Xylene (total)	5U	

1A
VOLATILE ORGANIC ANALYSIS DATA SHEET

SAMPLE NO.

Lab Name: BIOSPHERICS INCORPORATED Contract: ARGONNE

EBR II NO1

Lab Code: EBR II NO1 Case No. 93030806 SAS No.: _____ SDG No.: _____

Matrix: (soil/water) WATER Lab Sample ID: 93030806-2

Sample wt/vol: _____ (g/mL) Lab File ID: 8240

Level: (low/med) LOW Date Received: 03/03/93

% Moisture: not dec. _____ Date Analyzed: 03/08/93 ¹⁰ *mlt* *3/3K6*

GC Column: _____ ID: _____ (mm) Dilution Factor: _____

Soil Extract Volume: _____ (uL) Soil Aliquot Volume: 1 (uL)

CONCENTRATION UNITS:

CAS NO. COMPOUND (ug/L or ug/Kg) UG/L Q

97-63-2	Ethyl Methacrylate	5 <u>U</u>
107-02-8	Acrolein	50 <u>U</u>
108-05-4	Vinyl Acetate	10 <u>U</u>
107-13-1	Acrylonitrile	<i>mlt</i> 50 <u>U</u>
110-75-8	2-Chloroethyl Vinyl Ether	<i>3/3/96</i> 10 <u>U</u>
108-38-3	M & P Xylene	10 <u>U</u>
95-47-6	O Xylene	5 <u>U</u>
1634-04-4	Methyl tert-Butyl Ether	5 <u>U</u>
17-05-1	Allyl Chloride	50 <u>U</u>
126-98-7	Methacrylonitrile	50 <u>U</u>
107-12-0	Propanenitrile	50 <u>U</u>
74-88-4	Iodomethane	50 <u>U</u>
80-62-6	Methyl Methacrylate	20 <u>U</u>

FORM I VOA

Form by WindowsChem (707)664-0845;p/al1014/v3.0;1/1/96

OLM02.0

1A
VOLATILE ORGANIC ANALYSIS DATA SHEET

SAMPLE NO.

Lab Name: BIOSPHERICS INCORPORATED Contract: ARGONNE

Lab Code: EBRII NO2 Case No. 93030806 SAS No.: _____ SDG No.: _____

Matrix: (soil/water) WATER Lab Sample ID: 93030806-3

Sample wt/vol: _____ (g/mL) Lab File ID: 8240

Level: (low/med) LOW Date Received: 03/03/93

% Moisture: not dec. _____ Date Analyzed: 03/09/93 ¹⁰ *ML*

GC Column: _____ ID: _____ (mm) Dilution Factor: 1 *313 RL*

Soil Extract Volume: _____ (uL) Soil Aliquot Volume: _____ (uL)

CAS NO.	COMPOUND	CONCENTRATION UNITS:	
		(ug/L or ug/Kg)	UG/L
74-87-3	Chloromethane	10	U
74-83-9	Bromomethane	10	U
75-01-4	Vinyl Chloride	10	U
75-00-3	Chloroethane	10	U
75-09-2	Methylene Chloride	5	U
67-64-1	Acetone	10	U
75-15-0	Carbon Disulfide	5	U
75-35-4	1,1-Dichloroethene	5	U
75-34-3	1,1-Dichloroethane	5	U
540-59-0	1,2-Dichloroethene (total)	NR	
67-66-3	Chloroform	5	U
107-06-2	1,2-Dichloroethane	5	U
78-93-3	2-Butanone	100	U
71-55-6	1,1,1-Trichloroethane	5	U
56-23-5	Carbon Tetrachloride	5	U
75-27-4	Bromodichloromethane	5	U
78-87-5	1,2-Dichloropropane	5	U
10061-01-5	cis-1,3-Dichloropropene	5	U
79-01-6	Trichloroethene	5	U
124-48-1	Dibromochloromethane	5	U
79-00-5	1,1,2-Trichloroethane	5	U
71-43-2	Benzene	5	U
10061-02-6	trans-1,3-Dichloropropene	5	U
75-25-2	Bromoform	5	U
108-10-1	4-Methyl-2-Pentanone	10	U
591-78-6	2-Hexanone	10	U
127-18-4	Tetrachloroethene	5	U
79-34-5	1,1,2,2-Tetrachloroethane	5	U
108-88-3	Toluene	5	U
108-90-7	Chlorobenzene	5	U
100-41-4	Ethylbenzene	5	U
100-42-5	Styrene	5	U
1330-20-7	Xylene (total)	5	U

1A
VOLATILE ORGANIC ANALYSIS DATA SHEET

SAMPLE NO.

Lab Name: BIOSPHERICS INCORPORATED Contract: ARGONNE

EBR II NO2

Lab Code: EBR II NO2 Case No. 93030806 SAS No.: _____ SDG No.: _____

Matrix: (soil/water) WATER Lab Sample ID: 93030806-3

Sample wt/vol: _____ (g/mL) Lab File ID: 8240

Level: (low/med) LOW Date Received: 03/03/93

% Moisture: not dec. _____ Date Analyzed: 03/09/93 ¹⁰ *ml* _{3/3/96}

GC Column: _____ ID: _____ (mm) Dilution Factor: 1

Soil Extract Volume: _____ (uL) Soil Aliquot Volume: _____ (uL)

CAS NO. COMPOUND CONCENTRATION UNITS:
(ug/L or ug/Kg) UG/L Q

97-63-2	Ethyl Methacrylate	5 U	
107-02-8	Acrolein	50 U	
108-05-4	Vinyl Acetate	10 U	
107-13-1	Acrylonitrile	50 U	
110-75-8	2-Chloroethyl Vinyl Ether	10 U	
108-38-3	M & P Xylene	10 U	
95-47-6	O Xylene	5 U	
1634-04-4	Methyl tert-Butyl Ether	5 U	
17-05-1	Allyl Chloride	50 U	
126-98-7	Methacrylonitrile	50 U	
107-12-0	Propanenitrile	50 U	
74-88-4	Iodomethane	50 U	
80-62-6	Methyl Methacrylate	20 U	

FORM I VOA

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1A
VOLATILE ORGANIC ANALYSIS DATA SHEET

SAMPLE NO.

TRIP BLANK

Lab Name: BIOSPHERICS INCORPORATED Contract: ARGONNE

Lab Code: BLANK Case No. 93030806 SAS No.: _____ SDG No.: _____

Matrix: (soil/water) WATER Lab Sample ID: 93030806-4

Sample wt/vol: _____ (g/mL) Lab File ID: 8240

Level: (low/med) LOW Date Received: 03/03/93

% Moisture: not dec. _____ Date Analyzed: 03/09/93 *mt*

GC Column: _____ ID: _____ (mm) Dilution Factor: 1 *313196*

Soil Extract Volume: _____ (uL) Soil Aliquot Volume: _____ (uL)

CAS NO.	COMPOUND	CONCENTRATION UNITS:	
		(ug/L or ug/Kg)	UG/L
74-87-3	Chloromethane		10 ¹ U
74-83-9	Bromomethane		10 ¹ U
75-01-4	Vinyl Chloride		10 ¹ U
75-00-3	Chloroethane		10 ¹ U
75-09-2	Methylene Chloride		5 ¹ U
67-64-1	Acetone		10 ¹ U
75-15-0	Carbon Disulfide		5 ¹ U
75-35-4	1,1-Dichloroethene		5 ¹ U
75-34-3	1,1-Dichloroethane		5 ¹ U
540-59-0	1,2-Dichloroethene (total)		5 ¹ U
67-66-3	Chloroform		NR
107-06-2	1,2-Dichloroethane		5 ¹ U
78-93-3	2-Butanone		100 ¹ U
71-55-6	1,1,1-Trichloroethane		5 ¹ U
56-23-5	Carbon Tetrachloride		5 ¹ U
75-27-4	Bromodichloromethane		5 ¹ U
78-87-5	1,2-Dichloropropane		5 ¹ U
10061-01-5	cis-1,3-Dichloropropene		5 ¹ U
79-01-6	Trichloroethene		5 ¹ U
124-48-1	Dibromochloromethane		5 ¹ U
79-00-5	1,1,2-Trichloroethane		5 ¹ U
71-43-2	Benzene		5 ¹ U
10061-02-6	trans-1,3-Dichloropropene		5 ¹ U
75-25-2	Bromoform		5 ¹ U
108-10-1	4-Methyl-2-Pentanone		10 ¹ U
591-78-6	2-Hexanone		10 ¹ U
127-18-4	Tetrachloroethene		5 ¹ U
79-34-5	1,1,2,2-Tetrachloroethane		5 ¹ U
108-88-3	Toluene		5 ¹ U
108-90-7	Chlorobenzene		5 ¹ U
100-41-4	Ethylbenzene		5 ¹ U
100-42-5	Styrene		5 ¹ U
1330-20-7	Xylene (total)		5 ¹ U

1A
VOLATILE ORGANIC ANALYSIS DATA SHEET

SAMPLE NO.

Lab Name: BIOSPHERICS INCORPORATED Contract: ARGONNE TRIP BLANK

Lab Code: BLANK Case No. 93030806 SAS No.: _____ SDG No.: _____

Matrix: (soil/water) WATER Lab Sample ID: 93030806-4

Sample wt/vol: _____ (g/mL) Lab File ID: 8240

Level: (low/med) LOW Date Received: 03/03/93

% Moisture: not dec. _____ Date Analyzed: 03/09/93 3/3/96

GC Column: _____ ID: _____ (mm) Dilution Factor: 1

Soil Extract Volume: _____ (uL) Soil Aliquot Volume: _____ (uL)

CAS NO. COMPOUND CONCENTRATION UNITS:
(ug/L or ug/Kg) UG/L 0

97-63-2	Ethyl Methacrylate	5 U
107-02-8	Acrolein	50 U
108-05-4	Vinyl Acetate	10 U
107-13-1	Acrylonitrile	50 U
110-75-8	2-Chloroethyl Vinyl Ether	10 U
108-38-3	M & P Xylene	10 U
95-47-6	O Xylene	5 U
1634-04-4	Methyl tert Butyl Ether	5 U
17-05-1	Allyl Chloride	50 U
126-98-7	Methacrylonitrile	50 U
107-12-0	Propanenitrile	50 U
74-88-4	Iodomethane	50 U
80-62-6	Methyl Methacrylate	20 U

FORM I VOA

Form by WinDocChem(707)864-0805/p/n11014/v3.0:1/1/96

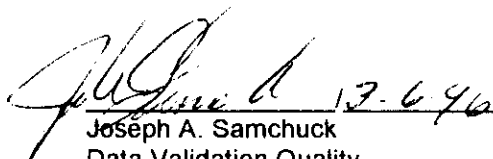
OLM02.0

Data Limitations and Validation Report
for Environmental Groundwater Samples
Collected from the Argonne National Laboratory - West
Scoville, ID
Case No. 93030806
SDG. No. 93030806
Target Analyte List (TAL) Metals plus Tin less Cyanide
Three Aqueous Samples

Validated by:


Ricky C. DePaul
Data Validation
Reviewer

Approved by:


Joseph A. Samchuck
Data Validation Quality
Assurance Officer

A. TITLE:

INORGANIC DATA LIMITATIONS and VALIDATION REPORT

Project Site: Waters from Argonne National Laboratory - West
Sample Type: Aqueous samples
Analysis Type: TAL Metals plus Tin less Cyanide
Case No.: 93030806
SDG. No.: 93030806

B. INTRODUCTION:

A complete review, following the procedures outlined in SMO-SOP-12.1.5¹, was performed on the data package, labeled Case No. 93030806, SDG# 93030806, submitted by Biospherics Incorporated. Based upon the information available for review, it appears as though, the laboratory analyzed the aforementioned water samples from the Argonne National Laboratory - West according to SW846 analytical protocols. The deliverable format does not comply with data package requirements pursuant with Level A validation protocol. The review could not include Level A data validation confirmation.

C. CONTRACT AND TECHNICAL REVIEW:

Site: Water from Argonne National Laboratory - West
Type: TAL Metals plus Tin less Cyanide
Case No.: 93030806
SDG No.: 93030806
Laboratory: Biospherics Incorporated

Sample Identification:

<u>FIELD ID</u>	<u>LAB ID</u>
MW-11	93030806-1
EBR II NO1	93030806-2
EBR II NO2	93030806-3

CTR COMMENTS:

1. The validator attempted to verify the reported result for sodium as noted for sample MW-11. The data package raw data sheets were legible and lended themselves to this purpose. The laboratory performed a 10X dilution for this analyte and chose to report the original undiluted sample result. The validator has no way of verifying the correct reporting procedure for this analyte in this sample in the absence of results for the ICP linear range analysis and holding times as omitted from the raw data and the Form XIVs. It appears as though the result may be reported as a valid and legitimate number. However, numerous other problems were noted with reported sample results to introduce enough question and uncertainty regarding data usability to render the data nonusable. Another specific example is detailed below:

The laboratory misrepresents sample results and incorrectly reports values for mercury as noted in all three samples. Specifically, the laboratory reported positive values for mercury in samples EBR-II NO1 and EBR II NO2. The raw data indicates that these sample data were in fact negative or below the detection limit results which should have been reported to < 0.5 ug/L, i.e., 0.5 U ug/L. This was not done. This type of rudimentary reporting inconsistency pervades much of the data which was subject to close scrutiny via validation Level A review. It is the professional opinion of the data reviewer that such inconsistencies severely compromise sample data quality to a degree which renders this data nonusable.

2. The validator reviewed Graphite Furnace Atomic Absorption (GFAA) raw data for selenium and noted numerous dilution analyses. The validator specifically looked at sample MW-11 regarding the positively reported result for this analyte in this sample. The Form XIV did not assist in ascertaining which analysis should have been used for reporting purposes. Dilution factors were not reported as is necessary on the Form XIV. The laboratory reported 5 ug/L for selenium in this sample. However, the raw data indicated a nondetected (negative) result for this analyte in each of the dilution analyses. Thus, it is the professional opinion of the data reviewer that extreme uncertainty exists as to the proper reporting of selenium in this sample. This result should not be used for quantitative purposes as the data reviewer is unable to verify sample calculation for selenium as stipulated and required for Level A validation protocol.
3. Additional problems were noted regarding improper reporting of the lead sample result as noted in sample MW-11. Again the laboratory reports a positive value for lead at 5 ug/L; whereas, the raw data for lead as noted for this sample was listed at < 5 ug/L.

A similar problem was noted regarding arsenic as noted in sample MW-11. Again the laboratory reported a positive value of 5 ug/L for this analyte in this sample. However, the GFAA raw data for this analyte indicated a nondetected result of < 5 ug/L, i.e., 5.0 U ug/L.

Additionally, the laboratory incorrectly reported the instrument response level for thallium as noted in sample MW-11 which is inherently wrong. The laboratory should have correctly reported a final analyte concentration of 5.0 U ug/L. To reiterate, these are the types of reporting inconsistencies which have been routinely encountered with these data packages and it is these types of problems which render the data unusable as reported. *These results should not be used for quantitative purposes as these improper reporting practices artificially bias the sample data set and adversely impact statistical interpretation of the sample data.*

4. Chain-of-Custody Forms were not provided for the samples in this SDG. Hence, the sample data could not be adequately analyzed for holding time and sample preservation requirements.
5. Portions of the raw data were illegible and provided no useful information. This anomaly is noted here for completeness.
6. A Continuing Calibration Verification (CCV) Percent Recovery (%R) for arsenic was below the 90% quality control limit.
7. The sample data were not evaluated for blank contamination. The laboratory reported negative results on Form I's and associated quality control data. Dilution factors were not chronicled on the Form XIVs as necessary for proper evaluation of this parameter. The absence of reporting limits and the practice of reporting results below SOW reporting limits compromised the blank evaluation process. The data reviewer could not therefore evaluate blank contamination in accordance with document SMO-SOP-12.1.5¹.
8. The laboratory did not adequately complete the ICP Interference Check Sample (ICS) Form IV. One of the purposes of analyzing the ICP ICS solution is to determine the potential impact of the four interfering analytes on potentially impacted analytes. This was not done. The interfering analytes aluminum, calcium, and magnesium were noted reported for this solution.

The ICSAB recovery for zinc exceeded the 120% upper quality control limit. It is noted that interfering analytes were present in the environmental samples at sufficiently low levels as to not introduce interference affects.

9. The Matrix Spike (MS) Form 5A and laboratory duplicate Form 6 were erroneously reported with numerous reporting inconsistencies rendering them useless without major revisional incorporations. Based upon the severity and extent of the aforementioned problems as noted above, it is in the professional opinion of the data reviewer that evaluation of the matrix spike and duplicate quality

control parameters will not increase data quality. Hence, the sample data were not further evaluated for these parameters.

10. The aqueous Laboratory Control Sample (LCS) recoveries for numerous analytes were not reported in some instances. Thus, this quality control parameter provides no useful information regarding data usability.

D. DATA LIMITATION OVERVIEW:

a. Summary of Qualified Data

Sample MW-11 could not be fully evaluated given the limitations of the data package deliverable. Sample data qualifications were not made for the aforementioned quality control noncompliances (anomalies) as it is not possible to ascertain a cumulative affect of the type or severity of problems impacting sample data quality based upon the unacceptable format of the data package deliverable.

Sample EBR II NO1 could not be fully evaluated given the limitations of the data package deliverable. Sample data qualifications were not made for the aforementioned quality control noncompliances (anomalies) as it is not possible to ascertain a cumulative affect of the type or severity of problems impacting sample data quality based upon the unacceptable format of the data package deliverable.

Sample EBR II NO2 could not be fully evaluated given the limitations of the data package deliverable. Sample data qualifications were not made for the aforementioned quality control noncompliances (anomalies) as it is not possible to ascertain a cumulative affect of the type or severity of problems impacting sample data quality based upon the unacceptable format of the data package deliverable.

E. LABORATORY APPRAISAL:

The data package was presented in a format which could not be fully evaluated as per the validation review requirements as defined by Level A validation review criteria. Qualifications applied to the data serve to indicate problems which could effectively be identified based upon specific noncompliant quality control parameters. Additionally, various anomalies and inconsistencies prevented a logical and systematic evaluation process of identifying an qualifying analytical results with a given amount of certainty. The following notable items illustrate the systematic problems associated with this deliverable:

- inconsistent reporting of analytical results (i.e., results reported both above and below detection limits referenced in the SOW).
- Absence of laboratory qualifications.
- Omissions of various analytes on various quality control summary forms (i.e., percent recoveries).

Furthermore, deficiencies noted with data presentation and reporting may not preclude additional, more severe problems with the data which could in affect render the data nonusable. It is not possible to make an accurate and complete assessment of the data as described above. Furthermore, overall data usability cannot be appraised for this data set as a result of problems noted with the deliverable.

F. REFERENCES:

1. Standard Operating Procedure For Inorganic Data Validation, "SMO-SOP-12.1.5", Environmental Restoration Program, EG&G, Inc., 1991.

APPENDIX A

RESULTS AS REPORTED BY THE LABORATORY

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INORGANIC ANALYSIS DATA SHEET

SAMPLE NO.

MW-11

Lab Name: BIOSPHERICS INCORPORATED Contract: ARGONNE

Lab Code: 93030806 Case No.: 93030806 SAS No.: _____ SDG No.: _____

Matrix (soil/water): WATER Lab Sample ID: 93030806-1

Level (low/med): LOW Date Received: 03/08/93

% Solids: 0.0

Concentration Units (ug/L or mg/kg dry weight): UG/L

CAS No.	Analyte	Concentration	C	Q	M
7429-90-5	Aluminum	-75			P
7440-36-0	Antimony	19.5			P
7440-38-2	Arsenic	5			F
7440-39-3	Barium	34.9			P
7440-41-7	Beryllium	0.1			P
7440-43-9	Cadmium	2.3			P
7440-70-2	Calcium	19093			P
7440-47-3	Chromium	7.8			P
7440-48-4	Cobalt	-0.1			P
7440-50-8	Copper	5			P
7439-89-6	Iron	71			P
7439-92-1	Lead	5			F
7439-95-4	Magnesium	11634			P
7439-96-5	Manganese	-1.9			P
7439-97-6	Mercury	-0.028			CV
7440-02-0	Nickel	-1.5			P
7440-09-7	Potassium	3200			A
7782-49-2	Selenium	5			F
7440-22-4	Silver	1.3			P
7440-23-5	Sodium	17193			P
7440-28-0	Thallium	-0.9			F
7440-62-2	Vanadium	9.4			P
7440-66-6	Zinc	7.2			P
	Cyanide				
7440-31-5	Tin	-13.4			P

Color Before: _____ Clarity Before: _____ Texture: _____

Color After: _____ Clarity After: _____ Artifacts: _____

Comments: _____

U.S. EPA - CLP
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INORGANIC ANALYSIS DATA SHEET

SAMPLE NO.

EBR II NO1

Lab Name: BIOSPHERICS INCORPORATED Contract: ARGONNE

Lab Code: 93030806 Case No.: 93030806 SAS No.: _____ SDG No.: _____

Matrix (soil/water): WATER Lab Sample ID: 93030806-2

Level (low/med): LOW Date Received: 03/08/93

% Solids: 0.0

Concentration Units (ug/L or mg/kg dry weight): UG/L

CAS No.	Analyte	Concentration	C	Q	M
7429-90-5	Aluminum	8			P
7440-36-0	Antimony	3.9			P
7440-38-2	Arsenic	3			F
7440-39-3	Barium	38.6			P
7440-41-7	Beryllium	0.4			P
7440-43-9	Cadmium	0.2			P
7440-70-2	Calcium	19096			P
7440-47-3	Chromium	1.9			P
7440-48-4	Cobalt	0.7			P
7440-50-8	Copper	2.4			P
7439-89-6	Iron	24			P
7439-92-1	Lead	0.1			F
7439-95-4	Magnesium	11745			P
7439-96-5	Manganese	-1.7			P
7439-97-6	Mercury	0.134			CV
7440-02-0	Nickel	-4.4			P
7440-09-7	Potassium	3230			A
7782-49-2	Selenium	-0.146			F
7440-22-4	Silver	1.7			P
7440-23-5	Sodium	17402			P
7440-28-0	Thallium	0.8			F
7440-62-2	Vanadium	7.1			P
7440-66-6	Zinc	3.6			P
	Cyanide				
7440-31-5	Tin	-15.1			P

Color Before: _____ Clarity Before: _____ Texture: _____

Color After: _____ Clarity After: _____ Artifacts: _____

Comments: _____

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INORGANIC ANALYSIS DATA SHEET

SAMPLE NO.

EBR II NO2

Lab Name: BIOSPHERICS INCORPORATED Contract: ARGONNE

Lab Code: 93030806 Case No.: 93030806 SAS No.: _____ SDG No.: _____

Matrix (soil/water): WATER Lab Sample ID: 93030806-3

Level (low/med): LOW Date Received: 03/08/93

% Solids: 0.0

Concentration Units (ug/L or mg/kg dry weight): UG/L

CAS No.	Analyte	Concentration	C	Q	M
7429-90-5	Aluminum	-94			P
7440-36-0	Antimony	3.7			P
7440-38-2	Arsenic	5			F
7440-39-3	Barium	32			P
7440-41-7	Beryllium	0.3			P
7440-43-9	Cadmium	0.5			P
7440-70-2	Calcium	19083			P
7440-47-3	Chromium	-0.5			P
7440-48-4	Cobalt	-3.1			P
7440-50-8	Copper	6.2			P
7439-89-6	Iron	11			P
7439-92-1	Lead	5			F
7439-95-4	Magnesium	11388			P
7439-96-5	Manganese	-1.8			P
7439-97-6	Mercury	0.026			CV
7440-02-0	Nickel	9			P
7440-09-7	Potassium	3180			A
7782-49-2	Selenium	5			F
7440-22-4	Silver	0.1			P
7440-23-5	Sodium	16789			P
7440-28-0	Thallium	5			F
7440-62-2	Vanadium	2.6			P
7440-66-6	Zinc	3.7			P
	Cyanide				
7440-31-5	Tin	-20.6			P

Color Before: _____ Clarity Before: _____ Texture: _____

Color After: _____ Clarity After: _____ Artifacts: _____

Comments:
